

**VALIDATION OF A NEW SOIL VOC SAMPLER: REVISION OF
ASTM PRACTICE D 6418, STANDARD PRACTICE FOR USING
THE DISPOSABLE EN CORE[®] SAMPLER FOR SAMPLING AND
STORING SOIL FOR VOLATILE ORGANIC ANALYSIS, AND
DEVELOPMENT OF A SUBSURFACE SAMPLING/STORAGE
DEVICE FOR VOC ANALYSIS**

Topical Report

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ABSTRACT

Soil sampling and storage practices for volatile organic analysis must be designed to minimize loss of volatile organic compounds (VOCs) from samples. The En Core[®] sampler is designed to collect and store soil samples in a manner that minimizes loss of contaminants due to volatilization and/or biodegradation. An American Society for Testing and Materials (ASTM) standard practice, D 6418, Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis, describes use of the En Core sampler to collect and store a soil sample of approximately 5 grams or 25 grams for volatile organic analysis.

To support the ASTM practice, a study was performed to estimate the precision of the performance of the 5-gram and 25-gram En Core samplers to store soil samples spiked with low concentrations of VOCs. This report discusses revision of ASTM Practice D 6418 to include information on the precision of the En Core devices and to reference an ASTM research report on the precision study.

This report also discusses revision of the ASTM practice to list storage at -12 ± 2 °C for up to 14 days and at 4 ± 2 °C for up to 48 hours followed by storage at -12 ± 2 °C for up to 5 days as acceptable conditions for samples stored in the En Core devices. Data supporting use of these storage conditions are given in an appendix to the practice and are presented in the research report referenced for the precision study. Prior to this revision, storage in the device was specified at 4 ± 2 °C for up to 48 hours.

The En Core sampler is designed to collect soil samples for VOC analysis at the soil surface. To date, a sampling tool for collecting and storing subsurface soil samples for VOC analysis does not exist. Development of a subsurface VOC sampling/storage device was initiated in 1999. This device, which is called the Accu Core sampler, is designed so that a soil sample can be collected below the surface using a penetrometer and transported to the laboratory for analysis in the same container. During the past year, prototype devices have been tested for their performance in storing soil samples containing low concentrations of VOCs. The Accu Core sampler testing is also described in this report.

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EXECUTIVE SUMMARY

Soil sampling and storage practices for volatile organic analysis must be designed to minimize loss of volatile organic compounds (VOCs) from samples. The En Core[®] sampler is designed to collect and store soil samples in a manner that minimizes loss of contaminants due to volatilization and/or biodegradation. An American Society for Testing and Materials (ASTM) standard practice, D 6418, Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis, describes use of the En Core sampler to collect and store a soil sample of approximately 5 grams or 25 grams for volatile organic analysis. The sampler, which consists of a coring body/storage chamber, O-ring sealed plunger, and O-ring sealed cap, is designed to collect and hold a soil sample during shipment to the laboratory for analysis. After the sample is collected in the En Core sampler, the coring body is sealed with a slide-on cap and immediately becomes a sample storage chamber.

To support the ASTM practice, four studies have been performed to evaluate the performance of the En Core sampler for storage of soil samples spiked with VOCs. The first study was conducted to evaluate the performance of the device to store soil samples spiked with VOCs at high-level concentrations of approximately 2,500 µg/Kg under various conditions. This analyte concentration in the soil was selected to limit the influence of the analytical method on the data. A second study was conducted to answer questions on the performance of the En Core sampler for storage of soil samples containing low-level (<200 µg/Kg) concentrations of VOCs. In this study, soil samples were spiked with concentrations of VOCs at approximately 100 µg/Kg and stored under various conditions prior to analysis. The third study was performed to generate data on the performance of the 25-gram En Core sampler to store soil samples spiked with EPA Method 1311, Toxicity Characteristic Leaching Procedure (TCLP), volatile organic analytes under various conditions for 14 days. The low-level performance data and TCLP analyte storage data are included in appendices to the ASTM practice, and the ASTM research report describing the high-level study is referenced in the practice. The fourth study was performed to estimate the precision of the performance of the 5-gram and 25-gram En Core samplers to store soil samples spiked with low concentrations of VOCs. This report discusses revision of ASTM Practice D 6418 to include information on the precision of the En Core devices and to reference an ASTM research report on the precision study.

To minimize loss of volatile compounds due to volatilization and/or biodegradation from the time of collection until analysis or preservation in the laboratory, the ASTM practice originally specified sample storage in the En Core device at 4±2 °C for up to 48 hours with an option for storage times beyond 48 hours at 4±2 °C or -12±2 °C, provided it could be shown that the longer storage time did not influence the concentrations of VOCs of interest or that the data generated by analysis of the samples met the data quality objectives (DQOs). This report discusses revision of D 6418 to list storage at 4±2 °C for up to 48 hours, at -12±2 °C for up to 14 days, and at 4±2 °C for up to 48 hours followed by storage at -12±2 °C for up to 5 days as acceptable conditions for samples

stored in the En Core devices. Data supporting use of these storage conditions are given in an appendix to the practice and are presented in the research report referenced for the precision study. The revised practice also gives the option for storage of samples in the En Core sampler at 4 ± 2 °C or -12 ± 2 °C for longer times than those listed above, provided it can be shown that the longer storage time used does not influence the concentrations of the VOCs of interest or that the data generated by analysis of the samples meet the DQOs.

In August 1998, a revision of ASTM D 4547, Standard Practice for Sampling Waste and Soils for Volatile Organics, was approved. This revision is titled “D 4547, Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds.” Because of continuing developments in the area of sampling and handling techniques for VOC analysis, a second revision of D 4547 has been proposed. Several draft revisions of the guide have been reviewed to ensure that use of the En Core device is included (and not excluded) as an option for sampling and storing soil for volatile organic analysis and to help ensure that the revision is technically sound. Activities related to achieving ASTM approval of a second revision of this standard are described in this report.

The En Core sampler is designed to collect soil samples for VOC analysis at the soil surface. To date, a sampling tool for collecting and storing subsurface soil samples for VOC analysis does not exist. Development of a subsurface VOC sampling/storage device was initiated in 1999. This device, which is called the Accu Core sampler, is designed so that a soil sample can be collected below the surface using a penetrometer and transported to the laboratory for analysis in the same container. During the past year, prototype devices have been tested for their performance in storing soil samples containing low concentrations of VOCs. This report describes the Accu Core sampler testing that has been performed.

INTRODUCTION

A major problem in sampling soil for volatile organic analysis is preservation of sample integrity during storage and shipment of soil samples to the laboratory. Soil sampling and storage practices for volatile organic analysis must be designed to minimize loss of volatile organic compounds (VOCs) due to volatilization and/or biodegradation. Laboratory data can grossly underestimate the actual VOC concentrations in a soil if great attention is not paid to sampling and handling techniques (Turrieff and Klopp 1995).

The En Core[®] sampling/storage device provides a simple means for sampling soil and holding a soil sample during shipment to the laboratory for VOC analysis (Vitale et al. 1999). This device has three components: (1) the coring body/storage chamber, which is volumetrically designed to collect and store either a soil sample of approximately 5 grams or 25 grams, (2) an O-ring sealed plunger for nondisruptive extrusion of the sample into an appropriate container for analysis or preservation, and (3) a slide-on cap having an O-ring seal and locking arm mechanism. A diagram of the En Core sampling/storage device is shown in Figure 1. The seals of the device are provided by three Viton[™] O-rings (Figure 1). The coring body/storage chamber, plunger, and cap of the En Core sampler are constructed of a glass-filled inert composite polymer, polyphthalamide (RTP), making the device chemically compatible with soil matrices and contaminants. The En Core sampler is disposable. It is certified as clean when received from the manufacturer and is not to be reused. The En Core sampler has two reusable stainless steel attachments. These are a T-handle, which is used to push the sampler into the soil for sample collection; and an extrusion tool, which attaches to the plunger for extrusion of the sample from the coring body/storage chamber. These are shown in Figure 2. Each En Core sampler is supplied with a protective moisture-proof bag for shipment to the laboratory.

Western Research Institute (WRI) developed an American Society for Testing and Materials (ASTM) standard practice for using the En Core device. This practice is D 6418, Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis (ASTM 2001). To support the ASTM practice, four studies have been performed to evaluate the performance of the En Core sampler for storage of soil samples spiked with VOCs. The first study was conducted to evaluate the performance of the device to store soil samples spiked with VOCs at high-level concentrations of approximately 2,500 µg/Kg under various conditions (Sorini and Schabron 1999a). This analyte concentration in the soil was selected to limit the influence of the analytical method on the data. A second study was conducted to answer questions on the performance of the En Core sampler for storage of soil samples containing low-level (<200 µg/Kg) concentrations of VOCs (Sorini et al. 2001 and 2002a). In this study, soil samples were spiked with concentrations of VOCs at approximately 100 µg/Kg and stored under various conditions prior to analysis. The third study was performed to generate data on the performance of the 25-gram En Core sampler to store soil samples spiked with EPA Method 1311, Toxicity Characteristic Leaching Procedure (TCLP) (U.S. EPA 1992), volatile organic analytes under various conditions for 14 days

(Sorini et al. 2001 and 2002a). The low-level performance data and TCLP analyte storage data are included in appendices to the ASTM practice (ASTM 2002a), and the ASTM research report describing the high-level study (Sorini and Schabron 1999b) is referenced in the practice. The fourth study was performed to estimate the precision of the performance of the 5-gram and 25-gram En Core samplers to store soil samples spiked with low concentrations of VOCs. ASTM Practice D 6418 was revised to include information on the precision of the En Core devices and to reference an ASTM research report on the precision study (Sorini et al. 2002c).

To minimize loss of volatile compounds due to volatilization and/or biodegradation from the time of collection until analysis or preservation in the laboratory, the ASTM practice (ASTM 2001 and 2002a) originally specified sample storage in the En Core device at 4 ± 2 °C for up to 48 hours with an option for storage times beyond 48 hours at 4 ± 2 °C or -12 ± 2 °C, provided it could be shown that the longer storage time did not influence the concentrations of VOCs of interest or that the data generated by analysis of the samples met the data quality objectives (DQOs). During revision of the practice to include the precision information, D 6418 was also revised to list storage at 4 ± 2 °C for up to 48 hours, at -12 ± 2 °C for up to 14 days, and at 4 ± 2 °C for up to 48 hours followed by storage at -12 ± 2 °C for up to 5 days as acceptable conditions for samples stored in the En Core devices. Data supporting use of these storage conditions are given in an appendix to the practice and are presented in the research report referenced for the precision study (Sorini et al. 2002c). The revised practice also gives the option for storage of samples in the En Core sampler at 4 ± 2 °C or -12 ± 2 °C for longer times than those listed above, provided it can be shown that the longer storage time does not influence the concentrations of the VOCs of interest or that the data generated by analysis of the samples meet the DQOs.

The revision of D 6418 to include precision information on the performance of the En Core samplers to store soil samples spiked with low concentrations of VOCs and to include storage at -12 ± 2 °C for up to 14 days and at 4 ± 2 °C for up to 48 hours followed by storage at -12 ± 2 °C for up to 5 days as acceptable conditions for samples stored in the En Core devices was balloted within ASTM Subcommittee D 34.01 on Sampling, Monitoring, and Characterization, ASTM Main Committee D 34 on Waste Management, and the ASTM Society. The revision passed balloting with no negative votes and is designated as D 6418-03 (ASTM 2003).

In August 1998, a revision of ASTM D 4547, Standard Practice for Sampling Waste and Soils for Volatile Organics, was approved. This revision is titled “D 4547, Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds” (ASTM 2002b). Because of continuing developments in the area of sampling and handling techniques for VOC analysis, a second revision of D 4547 has been proposed. Several draft revisions of the guide have been reviewed to ensure that the En Core sampler is fairly represented and to help ensure that the revision is technically sound. Work continues on achieving ASTM approval of a second revision of this standard.

The En Core sampler is designed to collect soil samples for VOC analysis at the soil surface. To date, a sampling tool for collecting and storing subsurface soil samples for VOC analysis does not exist. Development of a subsurface VOC sampling/storage device was initiated in 1999. This device, which is called the Accu Core sampler, is designed so that a soil sample can be collected below the surface using a penetrometer and transported to the laboratory for analysis in the same container. During the past year, prototype devices have been tested for their performance in storing soil samples containing low concentrations of VOCs.

This report describes revision and balloting of ASTM Practice D 6418 to include precision information on the performance of the En Core samplers to store soil samples spiked with low concentrations of VOCs and to include storage at -12 ± 2 °C for up to 14 days and at 4 ± 2 °C for up to 48 hours followed by storage at -12 ± 2 °C for up to 5 days as acceptable conditions for samples stored in the En Core devices. Activities related to the proposed revision of ASTM Guide D 4547 are also described, as well as Accu Core sampler prototype testing that has been performed.

OBJECTIVES

The overall objectives of this task are to facilitate national acceptance of the En Core device for sampling and storing soil for VOC analysis and to validate the performance of the Accu Core subsurface sampler for sampling and storing soil for VOC analysis. The activities that were performed over the last 12 months to help achieve these objectives are described below.

- ASTM Practice D 6418 was revised to include precision information on the performance of the En Core samplers to store soil samples spiked with low concentrations of VOCs and to include storage at -12 ± 2 °C for up to 14 days and at 4 ± 2 °C for up to 48 hours followed by storage at -12 ± 2 °C for up to 5 days as acceptable conditions for samples stored in the En Core devices.
- The revision of D 6418 was balloted within ASTM Subcommittee D 34.01 on Sampling, Monitoring, and Characterization, ASTM Main Committee D 34 on Waste Management, and the ASTM Society, and was approved.
- Activities involving reviewing and commenting on proposed draft revisions of D 4547, Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds, were performed.
- Prototype Accu Core samplers were tested for their performance in storing soil samples containing low concentrations of VOCs.

TECHNICAL APPROACH

Revision of ASTM Practice D 6418, Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis

As discussed, a study to estimate the precision of the performance of the 5-gram and 25-gram En Core samplers to store soil samples spiked with low concentrations of VOCs (Sorini et al. 2002c, 2002d, and 2003) was performed so information on the precision of the devices could be added to ASTM Practice D 6418. Table 1, which is presented in the reports on the precision study (Sorini et al. 2002c, 2002d, and 2003), shows a summary of the information that was obtained. This information was used to prepare the following precision statements for inclusion in the revision of D 6418. “The estimated percent relative standard deviations of low-level ($<200 \mu\text{g/Kg}$) concentrations of methylene chloride (MeCl_2), methyl tert-butyl ether (MTBE), 1,1-dichloroethane, cis-1,2-dichloroethylene (CDCE), chloroform, benzene, trichloroethylene (TCE), toluene, tetrachloroethylene (PCE), ethyl benzene, and o-xylene due to storage of spiked samples of two different soils in the 5-g and 25-g En Core samplers have been shown to range from 0% to 10% for the following conditions: 4 ± 2 °C for 48 hours, 4 ± 2 °C for seven days, -12 ± 2 °C for seven days, -12 ± 2 °C for 14 days, 4 ± 2 °C for 48 hours followed by -12 ± 2 °C for five days, and 4 ± 2 °C for 48 hours followed by -12 ± 2 °C for 12 days. For storage of samples at 4 ± 2 °C for 14 days, these values range from 0% to 14% for the compounds listed above. For vinyl chloride, the values range from 0% to 14% for all of the storage conditions.” Reference to the ASTM research report (Sorini et al. 2002c), which describes the precision study and is available from ASTM, was also included with this information.

As discussed, D 6418 originally specified sample storage in the En Core device at 4 ± 2 °C for up to 48 hours with an option for storage times beyond 48 hours at 4 ± 2 °C or -12 ± 2 °C, provided it could be shown that the longer storage time did not influence the concentrations of VOCs of interest or that the data generated by analysis of the samples met the data quality objectives (DQOs) (ASTM 2001 and 2002a). A second proposed change to D 6418 that was balloted along with the precision information was to list storage at -12 ± 2 °C for up to 14 days and at 4 ± 2 °C for up to 48 hours followed by storage at -12 ± 2 °C for up to 5 days as acceptable conditions for samples stored in the En Core samplers. Data supporting use of these storage conditions are presented in Appendix X1 of D 6418 (ASTM 2002a) and in the ASTM research report on the precision study (Sorini et al. 2002c). An option for storage of samples in the En Core sampler at 4 ± 2 °C or -12 ± 2 °C for longer times than those listed above, provided it can be shown that the longer storage time does not influence the concentrations of the VOCs of interest or that the data generated by analysis of the samples meet the DQOs, is also included in the revision.

The revision of D 6418 to include the precision information on the performance of the En Core samplers and additional storage conditions for samples stored in the devices was balloted for approval within ASTM Subcommittee D 34.01 on Sampling, Monitoring, and Characterization,

ASTM Main Committee D 34 on Waste Management, and the ASTM Society in early 2003. Balloting results were discussed at the ASTM D 34 Main Committee meeting in February 2003.

Proposed Revision of ASTM Guide D 4547, Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds

In August 1998, a revision of ASTM D 4547, ASTM's guidance document on sampling waste and soils for VOC's, was approved by ASTM. This revision is titled "D 4547, Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds" (ASTM 2002b). Because of continuing developments in the area of sampling and handling techniques for VOC analysis, a second revision of D 4547 has been proposed. Several draft revisions of the guide have been reviewed to ensure that the En Core sampler is fairly represented and to help ensure that the revision is technically sound. Changes to the guide that have been proposed and accepted by ASTM Subcommittee D 34.01 and Main Committee D 34 for the second revision include discussing freezing as a preservation technique and referencing D 6418 in the section of the guide that discusses sample storage in a coring device.

It has been proposed that a revision of D 4547 include reference to a technique for high-level analysis involving sample collection using a modified disposable plastic syringe from which the lower end with the needle attachment and the plunger cap has been removed. The sample is collected in the modified syringe and then immediately extruded into an empty volatile organic analysis (VOA) vial for storage of the sample during transportation to the laboratory. This technique is referred to as the "empty VOA vial method." Testing has been performed showing that extrusion of high-level samples into empty VOA vials can result in significantly greater VOC losses from samples as compared to losses resulting from sample extrusion directly into methanol (Sorini et al. 2002b). As a result, a statement has been included in the draft proposed revision of this document that states "having methanol present in the collection vessel reduces the possibility that VOCs could be lost during the transfer step (i.e., extrusion of a plug of soil from the sampling tool)."

Preliminary Tests to Evaluate the Performance of the Accu Core Sampler Prototypes

The Accu Core sampler consists of a cylindrical stainless steel sampler section that is designed to fit in a Geoprobe® dual-tube penetrometer soil sampling system, and two end caps having locking arms. Each end cap has a septum port and contains a Teflon®-lined natural rubber septum. When the stainless steel sampler section filled with soil is removed from the Geoprobe liner, the ends of the section are cleaned and immediately capped with the Teflon side of the septum in contact with the soil. The caps on the ends of the section are locked together for shipment to the laboratory for analysis. The Accu Core sampler is designed to collect a sample of approximately 25 grams of soil, based on an assumed soil density of 1.7 g/cm³.

Five sets of preliminary tests have been conducted to evaluate the performance of prototypes of the Accu Core sampler. The experimental design for each set of tests is described below.

First Set of Preliminary Tests

This set of tests was designed to evaluate the performance of the Accu Core samplers to store samples of mountain soil spiked with an aqueous solution containing twelve VOCs at low-level concentrations of <200 µg/Kg. The mountain soil contains 75% sand, 13% silt, 12% clay, and 4.3% organic material. This soil was characterized by the University of Wyoming Soils Laboratory using standard soil analytical methods and was homogenized prior to its characterization and use in the testing.

The VOCs used in the testing were vinyl chloride, methylene chloride (MeCl₂), methyl tert-butyl ether (MTBE), 1,1-dichloroethane, *cis*-1,2-dichloroethylene (CDCE), chloroform, benzene, trichloroethylene (TCE), toluene, tetrachloroethylene (PCE), ethyl benzene, and o-xylene. These compounds were selected as the analytes of interest because they are representative of halogenated and aromatic compounds that are of environmental concern.

To fill a sampler section for testing, soil was added to the sampler section using a spatula. The soil was added to the open end of the sampler section, which was fitted on the other end with a cap. The soil was compacted in the section using a stainless steel extrusion tool by patting the soil with the piston head of the tool. The end of the section to which soil was added was then capped, and the sampler section was turned over. The cap on the upper end of the sampler section was removed, and the above steps were repeated until a compacted soil plug filled the section. After a sampler section was filled, one end of the section was wiped clean and capped with a new cap. Then the open end of the sampler section was wrapped with aluminum foil. After all sampler sections were filled, the soil samples were stored in a walk-in cooler at a temperature of approximately 4 °C, so they would be cold when the low-level spiking was performed. This was done to minimize loss of the low concentrations of the volatile analytes during spiking.

The spiking solution used in the tests was prepared by adding 250 µL of gasoline to approximately 80 mL of VOC-free water in a 100-mL volumetric flask and diluting to volume. The mixture was stirred for 24 hours, and then the gasoline was separated from the water using a separatory funnel. A portion of the resulting gasoline-saturated water was added to a 40-mL VOA vial, and a methanol solution containing the compounds of interest, except for benzene and toluene, was injected into the gasoline-saturated water to give the spiking solution. The benzene and toluene present in the spiking solution came from the gasoline-saturated water. All of the solutions, including the spiking solution, were kept on ice to prevent loss of the volatile analytes.

The soil samples were spiked in the walk-in cooler at a temperature of approximately 4 °C. This minimized loss of the low concentrations of the volatile analytes during spiking. For spiking, the aluminum foil was removed from the soil-filled sampler section, the surface of the section was wiped clean, the spiking solution was injected into the middle of the soil plug through the open end of the section, and the section was immediately capped so that the two caps on the ends of the

section were locked together. The samples were spiked with 0.5 mL of spiking solution to give an approximate concentration of 100 µg/Kg of each analyte of interest in the samples, with the exception of benzene, toluene, ethylbenzene, and o-xylene. Because of their presence in the gasoline-saturated water, the concentrations of these analytes in the soil samples were expected to be greater than 100 µg/Kg. After all samples were spiked and capped, five random samples were extruded into 25 mL of methanol for analysis to give time-zero concentrations of the analytes of interest. Five of the remaining samples were randomly selected for storage at 4±2 °C for 48 hours. Because of problems in locking the sampler section caps, only three samples were available for storage at 4±2 °C for 48 hours followed by storage at -12±2 °C for 5 days. After the samples were held for the appropriate times, they were extruded into methanol for extraction and analysis. The methanol extracts of the samples were analyzed using guidance given in EPA Methods 5030 B (U.S. EPA 1996b) and 8260B (U.S. EPA 1996a).

To evaluate the data, the mean concentrations of the analytes of interest in the stored samples were compared to their mean concentrations in the time-zero samples by calculating average percent recovery. Before average percent recovery was calculated, the data sets were evaluated for outlier data points.

Second Set of Preliminary Tests

After the first set of tests was performed, minor design modifications were made to the Accu Core sampler. These changes involved extending the ledge on the locking arms, so the arms will lock more easily and securely, evenly spacing the notches in the cap to evenly position the sampler section against the Teflon liner on the septum to help with sealing, and polishing the edges of the stainless steel sampler sections to also help with sealing.

The second set of tests was conducted to evaluate the performance of the modified samplers and to evaluate sealing between the stainless steel sampler section and Teflon liner versus sealing between the stainless steel sampler section and the natural rubber side of the septum. In this testing, samples of the mountain soil were spiked with an aqueous solution containing the twelve VOCs used in the first set of tests at low-level concentrations of <200 µg/Kg. The sampler sections were filled using the procedure described for the first set of tests. After all sampler sections were filled, the soil samples were stored in the walk-in cooler at a temperature of approximately 4 °C, so they would be cold when the low-level spiking was performed.

Samples were prepared so there would be five time-zero samples with the Teflon side of the septum against the sample and sampler section edge (Teflon seal), five samples for storage at 4±2 °C for 48 hours with the Teflon side of the septum against the sample and sampler section edge (Teflon seal), and five samples for storage at 4±2 °C for 48 hours with the rubber side of the septum against the sample and sampler edge (rubber seal).

The spiking solution used in the testing was prepared and stored as described for the first set of tests. The soil samples were spiked in the walk-in cooler at a temperature of approximately 4 °C. For spiking, the aluminum foil was removed from the soil-filled sampler section, the surface of the section was wiped clean, the spiking solution was injected into the middle of the soil plug through the open end of the section, and the section was immediately capped so that the two caps on the ends of the section were locked together. To lessen the possibility of spiking solution being lost from the bottom of the sampler during the spiking procedure, the samples in this series of testing were spiked with 400 µL of spiking solution to give an approximate concentration of 70 µg/Kg of each analyte of interest in the samples, with the exception of benzene, toluene, ethylbenzene, and o-xylene. After all samples were spiked and capped, five random samples having the Teflon seals were extruded into 25 mL of methanol for analysis to give time-zero concentrations of the analytes of interest. The five remaining samples having Teflon seals and the five samples having rubber seals were stored at 4±2 °C for 48 hours. After the samples were held for the appropriate time, they were extruded into methanol for extraction and analysis. The methanol extracts of the samples were analyzed using guidance given in EPA Methods 5030 B (U.S. EPA 1996b) and 8260B (U.S. EPA 1996a).

To evaluate the data, the mean concentrations of the analytes of interest in the stored samples were compared to their mean concentrations in the time-zero samples by calculating average percent recovery. Before average percent recovery was calculated, the data sets were evaluated for outlier data points.

Third Set of Preliminary Tests

This set of tests was performed with En Chem, Inc. to evaluate the performance of the modified Accu Core samplers to store soil samples spiked with an aqueous solution containing the twelve VOCs used in the previous tests at low-level concentrations of <200 µg/Kg. The testing was performed by two laboratories to determine if similar results would be obtained. Sampler sections were filled with soil at WRI and En Chem. WRI sampler sections were filled with the mountain soil using the procedure described for the first set of tests. After the sampler sections were filled, the soil samples at both laboratories were stored in a walk-in cooler at a temperature of approximately 4 °C, so they would be cold when the low-level spiking was performed.

The spiking solution used in the testing was prepared and stored at both laboratories as described for the first set of tests. The soil samples were spiked at En Chem and WRI in walk-in coolers at a temperature of approximately 4 °C. The spiking solution was injected into the middle of the soil plug through the open end of the section, and the section was immediately capped so that the two caps on the ends of the section were locked together. For this testing, the volume of spiking solution was reduced from 500 µL and 400 µL used in the first and second set of tests, respectively, to 200 µL per sample as a precaution to ensure that the VOCs would not be lost from the soil at the bottom of the samplers prior to locking the caps on the sampler sections. The samples were spiked

to give an approximate concentration of 100 µg/Kg of each analyte of interest in the samples, with the exception of benzene, toluene, ethylbenzene, and o-xylene.

Fifteen samples were spiked at each laboratory (En Chem and WRI). After spiking, En Chem analyzed their five time-zero samples, and WRI analyzed their five time-zero samples. Five of the remaining samples were kept at each location for analysis after 48-hour storage at 4 ± 2 °C. The remaining five samples at En Chem were shipped by Federal Express to WRI for analysis after 48-hour storage at 4 ± 2 °C, and the remaining five samples at WRI were shipped by Federal Express to En Chem for analysis after 48-hour storage at 4 ± 2 °C.

After the samples were held for the appropriate times at each laboratory, they were extruded into methanol for extraction and analysis. The methanol extracts of the samples were analyzed using guidance given in EPA Methods 5030 B (U.S. EPA 1996b) and 8260B (U.S. EPA 1996a). To evaluate the data, the mean concentrations of the analytes of interest in the stored samples were compared to their mean concentrations in the appropriate time-zero samples by calculating average percent recovery. Before average percent recovery was calculated, the data sets were evaluated for outliers.

Fourth Set of Preliminary Tests

This set of tests was conducted with En Chem, Inc. to evaluate the performance of the Accu Core samplers to store samples of soil collected in the field and spiked with an aqueous solution containing the twelve VOCs used in the previous preliminary tests at low-level concentrations of <200 µg/Kg. Instead of filling the Accu Core sampler sections with soil in the laboratory, soil samples were collected in the sampler sections using the Geoprobe® dual-tube soil sampling system. It was believed that the soil collected in the field would be more compacted in the devices, and that this would result in higher VOC recoveries than those determined for the less compacted laboratory samples.

Thirty sampler sections were filled with uncontaminated soil in the field using the Geoprobe dual-tube penetrometer soil sampling system. Fifteen samples were shipped to WRI by Federal Express and 15 samples were shipped to En Chem by Federal Express. After sample collection, the sampler sections were sealed between wax paper, a rubber pad, and plywood. The pieces of plywood were screwed together to seal both ends of the sections. When the samples were received at WRI, they were stored as received in the walk-in cooler at a temperature of approximately 4 °C, so they would be cold when the low-level spiking was performed.

The spiking solution used in the testing was prepared and stored as described for the first set of tests. The soil samples were spiked in the walk-in cooler at a temperature of approximately 4 °C. For spiking, the soil-filled sampler sections were removed from the plywood. The surface of each section was wiped clean and a cap was placed on one end of each sample section. The spiking

solution was injected into the middle of the soil plug through the open end of the section, and the section was immediately capped. The samples were spiked with 200 μL of spiking solution to give an approximate concentration of 100 $\mu\text{g/Kg}$ of each analyte of interest in the samples, with the exception of benzene, toluene, ethylbenzene, and o-xylene.

The plan was for fifteen samples to be spiked by each laboratory (En Chem and WRI). After spiking, En Chem was to analyze their five time-zero samples, and WRI was to analyze their five time-zero samples. Five of the remaining samples were to be kept at each location for analysis after 48-hour storage at 4 ± 2 $^{\circ}\text{C}$. The remaining five samples at En Chem were to be shipped to WRI by Federal Express for analysis after 48-hour storage at 4 ± 2 $^{\circ}\text{C}$, and the remaining five samples at WRI were to be shipped to En Chem by Federal Express for analysis after 48-hour storage at 4 ± 2 $^{\circ}\text{C}$.

After the samples were held for the appropriate times at each laboratory, they were extruded into methanol for extraction and analysis. The methanol extracts of the samples were analyzed using guidance given in EPA Methods 5030 B (U.S. EPA 1996b) and 8260B (U.S. EPA 1996a). To evaluate the data, the mean concentrations of the analytes of interest in the stored samples were compared to their mean concentrations in the appropriate time-zero samples by calculating average percent recovery.

Fifth Set of Preliminary Tests

This set of tests was once again conducted with En Chem, Inc. to evaluate the performance of the Accu Core samplers to store samples of soil collected in the field and spiked with an aqueous solution containing the twelve VOCs used in the previous tests at low-level concentrations of <200 $\mu\text{g/Kg}$. In this testing, an attempt was made to address concerns raised by the results from the previous testing (fourth set of preliminary tests), in which VOCs were thought to be lost from the void space in the samplers when they were opened. The testing plan called for injection of 500 μL of methanol into the samples 24 hours prior to extrusion, so the void space would contain methanol, and when the samplers were opened for extrusion, the VOCs would be held in the methanol. Additional testing was also performed to compare analyte concentrations in contaminated soil samples stored in Accu Core samplers, En Core samplers, and empty VOA vials to analyte concentrations in contaminated soil samples immediately extruded into methanol in the field (time-zero samples).

Thirty sampler sections were filled with uncontaminated soil in the field using the Geoprobe dual-tube soil sampling system. After collection, the samples were spiked in a laboratory set up at the field site. The spiking solution that was used was prepared at WRI and shipped by Federal Express to the field site in a cooler with ice packs. The spiking solution was refrigerated until it was used for spiking. The solution was prepared as described for the previous tests. Thirty samples were spiked, 15 by WRI and 15 by En Chem, giving five time-zero samples, five samples for storage at 4 ± 2 $^{\circ}\text{C}$ for 48 hours, and five extra spiked samples for each laboratory. Spiking was performed as

previously described using a 200 μ L injection of spiking solution for each sample. After spiking, an attempt was made to inject 500 μ L of methanol into the time-zero samples prior to shipment to the laboratories. However, this was too large a volume of solvent to inject into the filled samplers, so the volume was reduced to 250 μ L. Despite the volume being reduced, methanol squirted out of the hole in the septum after the needle was removed. Various techniques were tried for methanol injection, such as injecting on a slant to get the methanol to the side of the sampler section and injection just below the septum on top of the sample, but none of these worked very well. Methanol squirted out of the hole in the septum in many cases, and often there was pooling on the septa around the hole after the squirting stopped. Similar problems were encountered for the stored samples.

Forty samples of contaminated soil were also collected in the field. Ten of these were in Accu Core sampler sections that were capped for storage of the samples; ten were subsampled using 5-gram En Core samplers; ten were subsampled using 5-gram Terra Core samplers (similar to cut-off syringe design) and immediately extruded into empty VOA vials; and ten were subsampled using 5-gram Terra Core samplers and immediately extruded into 2-oz jars containing 25-mL of methanol. Sample collection was alternated between the four collection procedures, so results would not be biased by collection order. Each set of ten samples was divided in half for shipment by Federal Express to WRI and En Chem for analysis.

RESULTS AND DISCUSSION

Revision of ASTM Practice D 6418, Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis

The revision of D 6418 to include the precision information on the performance of the En Core samplers and additional storage conditions for samples stored in the devices was approved by ASTM Subcommittee D 34.01 on Sampling, Monitoring, and Characterization, ASTM Main Committee D 34 on Waste Management, and the ASTM Society in early 2003. In the concurrent subcommittee/main committee balloting, the revision received 67 affirmative votes, no negative votes, and 86 abstentions. There was a 100% ballot return in the voting. As a result, the new revision is available from ASTM as a single standard and will be published in the ASTM Volume 11.04 later in 2003 (ASTM 2003).

In the balloting, there was one comment received that did not pertain to the revision, but rather to the composition of the En Core sampler. The comment was discussed with the person who submitted it, both over the telephone and at the D 34 Main Committee meeting. The author of the comment requested no changes to the standard based on his comment.

Proposed Revision of ASTM Guide D 4547, Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds

As mentioned, it has been proposed that a revision of D 4547 include reference to a technique for high-level analysis involving sample collection using a modified disposable plastic syringe. The sample is collected in the modified syringe and then immediately extruded into an empty VOA vial for storage of the sample during transportation to the laboratory. After the sample is received in the laboratory, methanol is injected through the septum of the VOA vial for methanol extraction and subsequent analysis of the extract. This technique is referred to as the “empty VOA vial method.”

The section in the most recently proposed revision of ASTM Guide D 4547 that describes this method received a negative vote during ASTM subcommittee/main committee balloting. The negative vote is based on testing results showing that extrusion of high-level samples into empty VOA vials can result in significantly greater VOC losses from samples as compared to losses resulting from sample extrusion directly into methanol (Sorini et al. 2002b). As a result, the following statement has been included in the section: “having methanol present in the collection vessel reduces the possibility that VOCs could be lost during the transfer step (i.e., extrusion of a plug of soil from the sampling tool).”

Preliminary Tests to Evaluate the Performance of the Accu Core Sampler Prototypes

First Set of Preliminary Tests

Performing the first set of preliminary tests showed that the caps on the ends of the sampler sections were difficult to lock together. For many of the samplers, when the locking arms on one side were snapped together, the arms on the other side would unsnap, and if these were once again snapped closed, the arms on the other side would unsnap. As previously mentioned, this problem resulted in five time-zero samples, five samples stored at 4 ± 2 °C for 48 hours, and three samples stored at 4 ± 2 °C for 48 hours followed by storage at -12 ± 2 °C for 5 days. It was also observed that the edges of the stainless steel sampler sections were not polished. The roughness of the edges of the sampler sections could interfere with the seal between the sampler section and Teflon liner on the rubber septum in the sampler cap. In addition, it was noticed that the notches in the sampler caps were not evenly spaced to evenly position the sampler section against the septa in the caps.

The results from the first set of preliminary tests are shown in Table 2. The average percent recovery values for the analytes from the stored samples as compared to their concentrations in the time-zero samples show that many of the VOCs were lost from the samples. It was believed that the problems discussed above contributed to the low VOC recoveries in this testing.

Second Set of Preliminary Tests

In performing the second set of preliminary tests, there was no difficulty in locking the caps onto the sampler sections. Extension of the ledge on the locking arms made this very easy to do.

Results from the second set of preliminary tests are shown in Table 3. For the time-zero samples, there was one sample for which the analyte concentration values were outliers. This was most likely due to an error in spiking the sample. As a result, values for four samples were used to determine the average analyte concentrations in the time-zero samples. As shown in Table 3, the percent relative standard deviation values for the four time-zero concentration values used to calculate the time-zero average concentration of each of the analytes are 8% or less, except for vinyl chloride. The value for vinyl chloride would be expected to be higher because of its high volatility. The low percent relative standard deviation values show that the four time-zero samples contained similar analyte concentrations, and that there were no problems in spiking these samples.

The average percent recovery values for the analytes of interest from the samples stored in the devices having the Teflon seal as compared to their concentrations in the time-zero samples show that the sampler sections with the Teflon seal lost VOCs. The average percent recovery values for the analytes of interest from the samples stored in the devices having the rubber seal also show low recovery values. However, it should be noted that the percent recovery for vinyl chloride is much higher for the rubber-sealed samples than for the Teflon-sealed samples, which indicates that a better seal was made with the rubber contact than with the Teflon contact. Rubber is known to absorb VOCs. This may account for the VOC loss shown for the samples in the devices having the rubber seals. Results from this series of tests raised concerns about the effectiveness of the sampler seal.

Third Set of Preliminary Tests

Results from WRI analysis of the WRI samples are shown in Table 4. For the WRI time-zero samples, there was one sample for which the analyte concentration values were outliers. As a result, values for four samples were used to determine the average analyte concentrations in the time-zero samples. As shown in Table 4, the percent relative standard deviation values for the four time-zero concentration values used to calculate the WRI time-zero average concentration of each of the analytes are 9% or less. This shows that the four time-zero samples contained similar analyte concentrations, and that there were no problems in spiking these samples. Also, the average percent recovery values for the analytes of interest from the stored samples prepared and analyzed by WRI as compared to their concentrations in the WRI time-zero samples indicate that the stored samples lost VOCs (Table 4).

The samples spiked by En Chem and sent to WRI for extrusion and analysis were difficult to extrude from the sampler sections because of their physical condition. They were very solid. One sample splashed such a significantly large volume of methanol out of the jar during extrusion that the analyte concentrations for the sample were not used to calculate average concentration of the analytes in the stored samples. Results from WRI analysis of the En Chem stored samples are shown in Table 5. The average percent recovery values for the analytes of interest from the stored samples that were spiked by En Chem and extruded and analyzed by WRI as compared to their

concentrations in the En Chem time-zero samples are significantly higher than the percent recovery values determined from WRI analysis of the WRI samples listed in Table 4. However, some of the values are lower than would be expected, indicating some VOC loss from the samples. As shown in Table 5, the percent relative standard deviation values for the five time-zero concentration values used to calculate the En Chem time-zero average concentration of each of the analytes are 13% or less, except for vinyl chloride at 21%. This shows that the five time-zero samples contained similar analyte concentrations, and that there were no problems in spiking these samples.

Results from En Chem analysis of the En Chem samples are shown in Table 6. The average percent recovery values for the analytes of interest from the stored samples prepared and analyzed by En Chem as compared to their concentrations in the En Chem time-zero samples are very similar to the values listed in Table 5 resulting from WRI's analysis of the En Chem stored samples.

Results from En Chem analysis of the WRI stored samples are shown in Table 7. The average percent recovery values for the analytes of interest from the stored samples prepared by WRI and analyzed by En Chem as compared to their concentrations in the WRI time-zero samples are very low and are similar to the values determined from WRI's analysis of the WRI stored samples (Table 4).

In this testing, the percent recovery values for the analytes of interest for both the En Chem and WRI samples were lower than would be expected. However, the values for the WRI samples were much lower than the values for the En Chem samples, suggesting a possible reason other than a sealing problem for the lower VOC recoveries. It was thought that perhaps the samples were not being compacted well enough in the sampler sections prior to spiking. This resulted in the fourth set of preliminary tests, in which samples for spiking were collected in the field.

Fourth Set of Preliminary Tests

The soil samples collected in the field in the Accu Core sampler sections were stored for approximately 10 days prior to testing. This long storage time caused the samples to harden and to be difficult to extrude from the sampler sections. One WRI time-zero sample was lost because a large volume of methanol splashed out of the jar when the sample was extruded because of the force with which the sample had to be pushed from the sampler section. It was also noted that one of the WRI stored samples was very different from the other stored samples in that it had head space within the sampler section.

The WRI data for the WRI stored samples showed very low analyte concentrations in the sample having void space. These data suggest that VOCs are lost from samples having head space in the Accu Core sampler sections when the samplers are opened for sample extrusion. The average percent recovery values calculated using the concentration values for each of the analytes of interest in the other four samples stored at WRI are shown in Table 8. The data presented in Table 8 show

acceptable percent recovery values for most of the analytes of interest. The values for TCE and PCE are lower. This is most likely not due to loss from the samples, but may be due to retention of these double-bonded compounds by the soil. The low percent recovery value for vinyl chloride is most likely due to loss during sample extrusion.

The stored En Chem samples were also difficult to extrude from the sampler sections. Splashing occurred when some of these were extruded. The average percent recovery values calculated using the concentration values for each of the analytes of interest in the five samples prepared by En Chem and analyzed by WRI are shown in Table 9. These values show trends similar to those shown by the data in Table 8. The average percent recovery values for TCE and PCE are lower than for the other compounds. Again, this does not appear to be due to loss from the samples. The data show acceptable recoveries of 80% or greater for the other analytes of interest.

In addition to the problem of the soil samples being difficult to extrude because of the long storage time, En Chem encountered mechanical problems with some of their equipment. Therefore, only WRI's data were evaluated for this series of testing. Results from WRI's testing suggest that VOCs can be lost from samples having void space when the sampler sections are opened after storage. This is a concern because the testing showed that when samples are collected in the field using the Accu Core samplers in the Geoprobe sampling system, the sampler sections are not uniformly filled and can contain head space in which VOCs will accumulate during sample storage. Loss of VOCs from head space in the sampler sections may explain some of the losses seen in the previous series of tests.

This series of tests involving sample collection in the field gave higher percent recovery values for the analytes of interest than those determined in the previous testing, which involved samples prepared in the laboratory. However, as mentioned, the samples used in the testing were very hard because they had been stored for 10 days prior to spiking. As a result, for these samples, there was much less of an opportunity for soil particles to make sealing the sampler sections difficult than for samples that are not hardened.

Fifth Set of Preliminary Tests

When the time-zero samples for the Accu Core spiked field samples were analyzed, they contained none of the analytes of interest above the analytical detection limits. It is believed that the analytes were lost when methanol injection into the samples was attempted. As a result, the stored samples were not analyzed, and no data were generated for this part of the testing. Results from analysis of the contaminated soil collected and stored in the prototype Accu Core samplers, En Core samplers, empty VOA vials, and methanol showed extremely high VOC levels in the soil samples. The concentrations were so high that the analytes served as a sample preservative, and no

information could be obtained on the performance of the different sampling/storage devices to hold the VOCs. Although no analytical data were obtained from this testing, collection of the prototype Accu Core samples in the field showed the need for a good sealing mechanism between the sampler sections and caps, because the edges of the sampler sections can be difficult to clean in the field. In addition, information was obtained that can be used in developing a technique for injecting methanol into the sampler sections prior to sample extrusion.

CONCLUSIONS

Progress was made toward further facilitating national acceptance of the En Core device through the activities performed during the last 12 months. ASTM Practice D 6418 was revised to include precision information on the performance of the En Core samplers and to include freezing as an acceptable storage condition for samples stored in En Core samplers. Activities involving reviewing and commenting on proposed draft revisions of D 4547 continued to help ensure that the En Core sampler is included in the guide and fairly represented, as well as helping to ensure that the guide provides technically correct information.

Data generated by the preliminary tests to evaluate the performance of the Accu Core sampler prototypes show that the sampler will hold VOCs in soil samples for analysis. However, the preliminary testing identified two issues that need to be addressed before the samplers can be put on the market and testing to validate their performance can be started. The first of these is that samples collected in the Accu Core samplers can contain head space in which VOCs will accumulate during storage. As a result, a technique to inject methanol into the samplers prior to opening them for sample extrusion must be developed. Secondly, the seal between the prototype Accu Core sampler cap and sampler section should be improved to insure that VOCs will not be lost during sample storage. In the field, the sampler sections are difficult to clean prior to capping, making an effective means of sealing the Accu Core samplers critical. Based on these findings, work continues on developing and testing a method to inject methanol into the samplers prior to sample extrusion and a sealing mechanism that will safeguard against loss of VOCs from samples during storage.

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Table 1. Estimated Percent Relative Standard Deviation of Low-Level VOC^a Concentrations^b Due to Storage of Spiked Soil Samples in En Core Samplers

Storage Condition	Estimated Percent Relative Standard Deviations of Concentration Values Due to Storage in the En Core Samplers			
	<u>5-g RBS^c</u>	<u>25-g RBS</u>	<u>5-g PS^d</u>	<u>25-g PS</u>

4±2 /C for 48 hrs.	0% - 5%	0% - 7%	0%	0% - 6%, except vinyl chloride at 13%; TCE at 10%; o-xylene at 8%
4±2 /C for 7 days	0% - 5%	0%	0% - 6%	0% - 7%, except vinyl chloride at 12%
4±2 /C for 14 days	5% - 13%	0% - 4%	0% - 5%, except o-xylene at 11%	0% - 14%
-12±2 /C for 7 days	0% - 3%	0%	0% - 3%	0% - 5%
-12±2 /C for 14 days	0% - 6%	0% - 4%	0%	0% - 4%
4±2 /C for 48 hrs. then -12±2 /C for 5 days	0% - 3%	0% -5%, except vinyl chloride at 14%	0%, except vinyl chloride at 10%; MeCl ₂ at 5%	0% - 5%
4±2 /C for 48 hrs. then -12±2 /C for 12 days	0% - 7%	0% - 6%	0% - 8%	0% - 6%

^a Analytes of interest are vinyl chloride, MeCl₂, MTBE, 1,1-dichloroethane, CDCE, chloroform, benzene, TCE, toluene, PCE, ethyl benzene, and o-xylene.

^b Concentrations of the VOCs in the samples were ~100 µg/Kg, except benzene concentrations were ~150 µg/Kg, toluene concentrations ranged from ~700 µg/Kg to ~1,400 µg/Kg, and o-xylene concentrations were ~200 µg/Kg.

^c River bank soil

^d Prairie soil

Table 2. Results from the First Set of Preliminary Tests on the Accu Core Sampler—Average Percent Recovery Values of Low-Level VOCs from Samples of Mountain Soil Stored in Accu Core Sampler Prototypes

Analyte	Time-Zero Conc. ^a	Storage at 4±2 /C for 48 hrs. Conc. ^a	Storage at 4±2 /C for 48 hrs./ 5 Days at -12±2 /C Conc. ^a
Vinyl Chloride	57	0.80 (1%) ^b	2.3 (4%) ^b
MeCl ₂	86	50 (58%)	35 (41%)

MTBE	85	82 (96%)	57 (67%)
1,1-Dichloro-ethane	83	47 (57%)	24 (29%)
CDCE	79	57 (72%)	32 (41%)
Chloroform	88	70 (80%)	56 (64%)
Benzene	187	129 (69%)	73 (39%)
TCE	73	59 (81%)	37 (51%)
Toluene	882	815 (92%)	662 (75%)
PCE	79	75 (95%)	47 (59%)
Ethylbenzene	146	156 (107%)	106 (73%)
o-Xylene	201	216 (107%)	143 (71%)

^a Units are µg/Kg.

^b Average percent recovery compared to time-zero concentrations

Table 3. Results from the Second Set of Preliminary Tests on the Accu Core Sampler – Average Percent Recovery Values of Low-Level VOCs from Samples of Mountain Soil Stored in Accu Core Sampler Prototypes Having Teflon® Seals Versus Rubber Seals

Analyte	Time-Zero Ave. Conc. ^a	48 hrs./4±2 °C Ave. Conc. ^c Teflon Seal	% Recovery with Teflon Seal	48 hrs./4±2 °C Ave. Conc. ^c Rubber Seal	% Recovery with Rubber Seal
Vinyl Chloride	41 (25%) ^b	0.4 (27%) ^d	1%	12 (23%) ^d	29%
MeCl ₂	73 (8%)	15 (21%)	21%	21 (25%)	29%

MTBE	71 (5%)	46 (32%)	65%	36 (42%)	51%
1,1-Dichloro-ethane	68 (7%)	27 (24%)	40%	35 (26%)	51%
CDCE	63 (4%)	32 (22%)	51%	32 (21%)	51%
Chloroform	77 (3%)	39 (24%)	51%	36 (30%)	47%
Benzene	100 (5%)	51 (24%)	51%	50 (27%)	50%
TCE	63 (3%)	40 (18%)	63%	36 (29%)	57%
Toluene	760 (3%)	555 (19%)		73%	430 (27%) 57%
PCE	72 (3%)	58 (18%)	81%	45 (22%)	62%
Ethylbenzene	133 (2%)	131 (13%)	98%	98 (21%)	74%
o-Xylene	182 (5%)	188 (14%)		103%	132 (18%) 72%

^a Average of analyte concentrations in four time-zero samples, µg/Kg

^b Percent relative standard deviation of the four time-zero concentration values used to calculate the average concentration value

^c Average of the analyte concentrations in five stored samples, µg/Kg

^d Percent relative standard deviation of the five stored sample concentration values used to calculate the average concentration value

Table 4. Results from the Third Set of Preliminary Tests on Accu Core Sampler Prototypes – Average Percent Recovery-WRI Data for WRI Samples

Analyte	Time-Zero Ave. Conc. ^a (WRI Data)	48 hrs./4±2 °C Ave. Conc. ^c (WRI Data)	% Recovery
Vinyl Chloride	30 (4%) ^b	<0.5 (0%) ^d	0%
MeCl ₂	89 (7%)	12 (29%)	13%
MTBE	96 (4%)	40 (27%)	42%

1,1-Dichloro-ethane	84 (7%)	25 (13%)	30%
CDCE	85 (8%)	30 (19%)	35%
Chloroform	98 (7%)	40 (17%)	41%
Benzene	52 (6%)	21 (16%)	40%
TCE	84 (5%)	42 (15%)	50%
Toluene	324 (4%)	178 (14%)	55%
PCE	89 (9%)	47 (15%)	53%
Ethylbenzene	133 (4%)	95 (10%)	71%
o-Xylene	164 (3%)	129 (9%)	79%

^a Average of analyte concentrations in four time-zero samples, µg/Kg

^b Percent relative standard deviation of the four time-zero concentration values used to calculate the average concentration value

^c Average of the analyte concentrations in five stored samples, µg/Kg

^d Percent relative standard deviation of the five stored sample concentration values used to calculate the average concentration value

Table 5. Results from the Third Set of Preliminary Tests on Accu Core Sampler Prototypes – Average Percent Recovery-WRI Data for Stored En Chem Samples

Analyte	Time-Zero Ave. Conc. ^a (En Chem Data)	48 hrs./4±2 °C Ave. Conc. ^c (WRI Data)	% Recovery
Vinyl Chloride	37 (21%) ^b	9.3 (71%) ^d	25%
MeCl ₂	74 (10%)	56 (16%)	76%
MTBE	82 (4%)	70 (12%)	85%
1,1-Dichloro-ethane	69 (8%)	50 (22%)	72%

CDCE	62 (11%)	49 (9%)	79%
Chloroform	65 (7%)	63 (11%)	97%
Benzene	83 (6%)	58 (13%)	70%
TCE	75 (13%)	52 (8%)	69%
Toluene	240 (0%)	292 (10%)	122%
PCE	61 (4%)	59 (8%)	97%
Ethylbenzene	82 (7%)	92 (6%)	112%
o-Xylene	88 (3%)	111 (7%)	126%

^a Average of analyte concentrations in five time-zero samples, µg/Kg

^b Percent relative standard deviation of the five time-zero concentration values used to calculate the average concentration value

^c Average of the analyte concentrations in four stored samples, µg/Kg

^d Percent relative standard deviation of the four stored sample concentration values used to calculate the average concentration value

Table 6. Results from the Third Set of Preliminary Tests on Accu Core Sampler Prototypes – Average Percent Recovery-En Chem Data for En Chem Samples

Analyte	Time-Zero Ave. Conc. ^a (En Chem Data)	48 hrs./4±2 °C Ave. Conc. ^c (En Chem Data)	% Recovery
Vinyl Chloride	37 (21%) ^b	12 (70%) ^d	32%
MeCl ₂	74 (10%)	56 (13%)	76%
MTBE	82 (4%)	85 (6%)	104%
1,1-Dichloro-ethane	69 (8%)	59 (15%)	85%
CDCE	62 (11%)	50 (10%)	81%

Chloroform	65 (7%)	58 (5%)	89%
Benzene	83 (6%)	68 (15%)	82%
TCE	75 (13%)	57 (13%)	76%
Toluene	240 (0%)	206 (9%)	86%
PCE	61 (4%)	52 (14%)	85%
Ethylbenzene	82 (7%)	73 (6%)	89%
o-Xylene	88 (3%)	79 (12%)	90%

^a Average of analyte concentrations in five time-zero samples, µg/Kg

^b Percent relative standard deviation of the five time-zero concentration values used to calculate the average concentration value

^c Average of the analyte concentrations in five stored samples, µg/Kg

^d Percent relative standard deviation of the five stored sample concentration values used to calculate the average concentration value

Table 7. Results from the Third Set of Preliminary Tests on Accu Core Sampler Prototypes – Average Percent Recovery–En Chem Data for Stored WRI Samples

Analyte	Time-Zero Ave. Conc. ^a (WRI Data)	48 hrs./4±2 °C Ave. Conc. ^c (En Chem Data)	% Recovery
Vinyl Chloride	30 (4%) ^b	0 (0%) ^d	0%
MeCl ₂	89 (7%)	0 (0%)	0%
MTBE	96 (4%)	32 (69%)	33%
1,1-Dichloro-ethane	84 (7%)	4.3 (198%)	5%
CDCE	85 (8%)	8.9 (143%)	10%
Chloroform	98 (7%)	14 (116%)	14%

Benzene	52 (6%)	7.4 (140%)	14%
TCE	84 (5%)	24 (60%)	29%
Toluene	324 (4%)	111 (44%)	34%
PCE	89 (9%)	34 (36%)	38%
Ethylbenzene	133 (4%)	70 (15%)	53%
o-Xylene	164 (3%)	92 (12%)	56%

^a Average of analyte concentrations in four time-zero samples, µg/Kg

^b Percent relative standard deviation of the four time-zero concentration values used to calculate the average concentration value

^c Average of the analyte concentrations in four stored samples, µg/Kg

^d Percent relative standard deviation of the four stored sample concentration values used to calculate the average concentration value

Table 8. Results from the Fourth Set of Preliminary Tests on Accu Core Sampler Prototypes – Average Percent Recovery-WRI Data for WRI Samples

Analyte	Time-Zero Ave. Conc. ^a (WRI Data)	48 hrs./4±2 °C Ave. Conc. ^c (WRI Data)	% Recovery
Vinyl Chloride	72 (10%) ^b	34 (9%) ^d	47%
MeCl ₂	93 (6%)	84 (11%)	90%
MTBE	89 (6%)	74 (10%)	83%
1,1-Dichloro-ethane	84 (5%)	73 (13%)	87%
CDCE	74 (4%)	61 (13%)	82%
Chloroform	92 (6%)	82 (12%)	89%
Benzene	43 (3%)	38 (12%)	88%

TCE	70 (4%)	46 (17%)	66%
Toluene	210 (6%)	164 (14%)	78%
PCE	57 (5%)	31 (15%)	54%
Ethylbenzene	107 (6%)	78 (11%)	73%
o-Xylene	129 (7%)	104 (13%)	81%

^a Average of analyte concentrations in four time-zero samples, µg/Kg

^b Percent relative standard deviation of the four time-zero concentration values used to calculate the average concentration value

^c Average of the analyte concentrations in four stored samples, µg/Kg (Values for sample having void space not included.)

^d Percent relative standard deviation of the four stored sample concentration values used to calculate the average concentration value

Table 9. Results from the Fourth Set of Preliminary Tests on Accu Core Sampler Prototypes – Average Percent Recovery-WRI Data for Stored En Chem Samples

Analyte	Time-Zero Ave. Conc. ^a (En Chem Data)	48 hrs./4±2 °C Ave. Conc. ^c (WRI Data)	% Recovery
Vinyl Chloride	47 (18%) ^b	40 (39%) ^d	85%
MeCl ₂	120 (10%)	121 (16%)	101%
MTBE	89 (12%)	82 (4%)	92%
1,1-Dichloro- ethane	63 (13%)	66 (17%)	105%
CDCE	60 (17%)	55 (18%)	92%
Chloroform	59 (13%)	71 (15%)	120%
Benzene	31 (18%)	26 (15%)	84%
TCE	63 (16%)	46 (15%)	73%

Toluene	116 (13%)	124 (14%)	107%
PCE	47 (17%)	30 (11%)	64%
Ethylbenzene	75 (19%)	79 (13%)	105%
o-Xylene	81 (16%)	103 (14%)	127%

^a Average of analyte concentrations in five time-zero samples, µg/Kg

^b Percent relative standard deviation of the five time-zero concentration values used to calculate the average concentration value

^c Average of the analyte concentrations in five stored samples, µg/Kg

^d Percent relative standard deviation of the five stored sample concentration values used to calculate the average concentration value

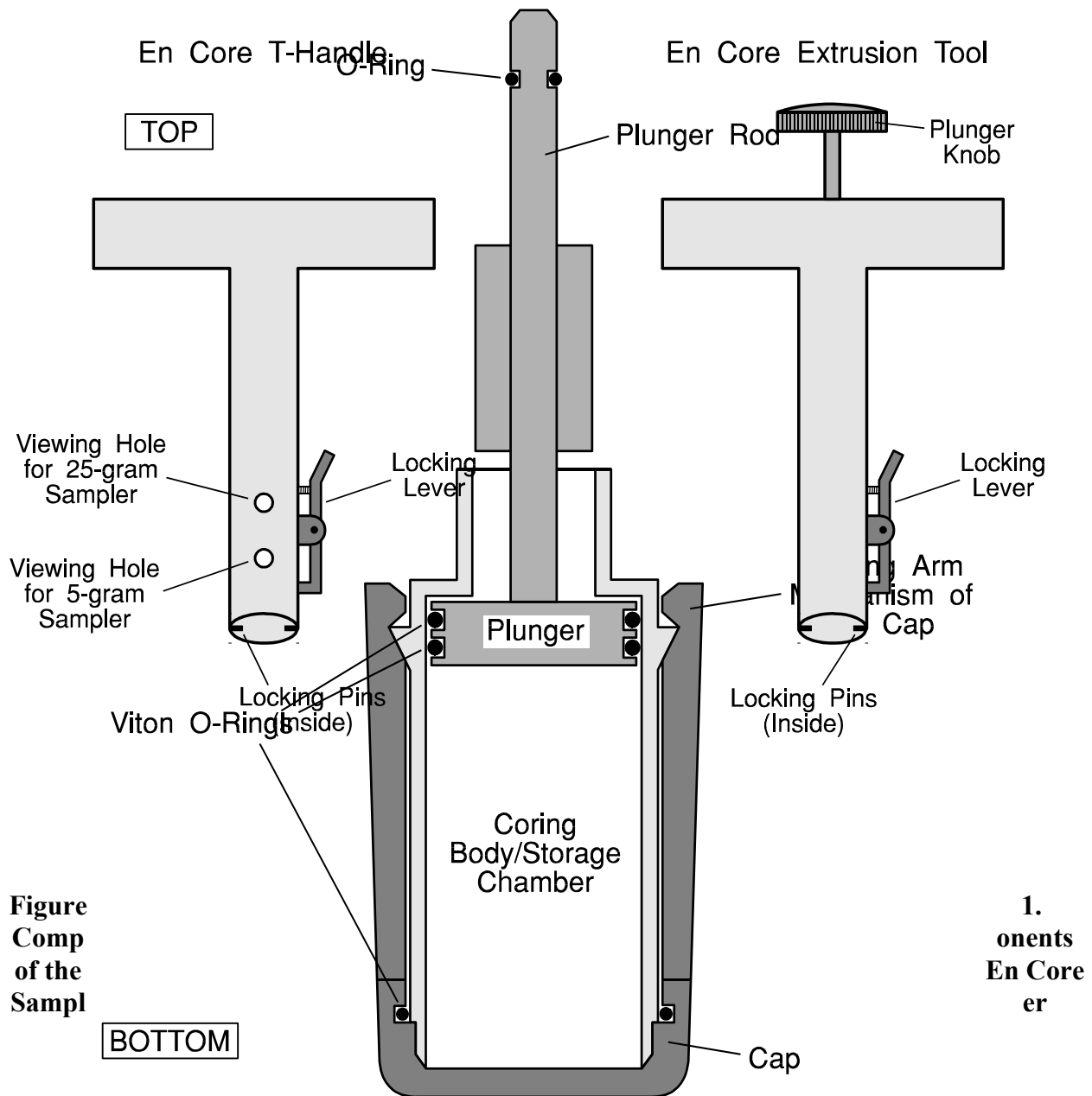


Figure 2. Reusable Attachments to the En Core Sampler